

## REMARKS

The Office Action was mailed in the present case on May 7, 2009, making a response due on or before August 7, 2009. This Response is being submitted, along with a Petition for Extension of Time Within the First Month, and the required extension fee of \$130.00 for a large entity. No additional fee is thought to be due at this time. If any additional fee is due for the continued prosecution of this application, please charge the same to Applicant's Deposit Account No. 50-2555 (Whitaker, Chalk, Swindle & Sawyer, LLP).

Applicant filed a Request For Continued Examination on April 27, 2009. In the amendment which accompanied that filing, Applicant had amended independent Claim 1 to call for a:

*"Calco-magnesian aqueous suspension having particles of solid matter with a solid matter content greater than or equal to 32% by weight, characterized in that it presents, before being put into suspension, a specific surface area, calculated according to the BET method, which is less than or equal to 10 m<sup>2</sup>/g."*

The Examiner had previously rejected Applicant's Claims 1-6 under 35 U.S.C. §103(a) as being unpatentable over the reference to *Huege et al.* (US 5616283). The Examiner argues that *Huege et al.* discloses a calco-magnesian suspension having particles of sold matter with a solid content of more than 39% by weight and a viscosity of less than 1000 cps (in fact,: "less than 2000 cps, preferably less than 1000 cps"). The Examiner also states that *Huege et al.* teaches a particles size that is finer than 20 mesh (which is approximately 841 µm) and is of the opinion that surface area is a function of particle size. This last conclusion leads the Examiner to conclude that the teaching of the reference thus anticipates the express language of Applicant's Claim 1, namely in describing "particles of solid matter with a specific surface area, calculated according to the BET method, which is less than or equal to 10 m<sup>2</sup>/g.

However, as Applicant has previously argued, even assuming that external specific surface area can be said to be a function of the particle size, it is clear that this is not the case with respect to the internal specific surface area. The external specific surface area is the Blaine specific surface area which is greatly affected by particles size changes and is more indicative of particle size (see the previously referenced U.S. Patent No. 4,829,107, column 1, lines 52-57, as well as column 4, line 31-40).

However, in Claim 1, the claimed feature is a "specific surface area less than or equal to 10 m<sup>2</sup>/g calculated according to the BET method" (Applicant's emphasis). As will be clear from the remarks which follow, this internal specific area is not dependent on the particle size.

Further, there is no specific surface area calculated according to the BET method disclosed in *Huege et al.* and in addition, no internal specific surface area can be derived from the disclosure of *Huege et al.* It is therefore clear that Claim 1 is not obvious over *Huege et al.* since this document only discloses that it is preferable to have a lime slurry with a very fine particle size to maintain the desired reactivity and to use a dispersing aid (alkali metal hydroxide) to further lower the viscosity of the slurry after the dispersing agent and the lime have been mixed, (see column 3, lines 50 to 55 and 57 to 67).

*Huege et al.* hence uses two additives to keep a low viscosity and also clearly does not disclose that with an internal specific surface area of less than or equal to 10 m<sup>2</sup>/g, without strictly requiring the use of a dispersant, a solid content greater than 32% by weight is obtained.

Further, as already mentioned, *Huege et al.* teaches the use of a particle size as fine as possible. A particle size as fine as possible will yield to an increased external specific surface area and therefore to an increased viscosity.

The taught particle size in *Huege et al.* is under 20 mesh (841  $\mu\text{m}$ ). The invention of the Applicant uses a  $d_{98}$  of less than 20  $\mu\text{m}$  which is about 42 times less. Therefore it would appear that the viscosity of a lime slurry prepared according to the teaching of Applicant's invention would lead to a viscosity which is drastically increased, since the particle size is strongly reduced. However, in actual practice, exactly the opposite effect is reached by following the teaching of the invention, because the internal specific surface area as claimed is radically different from the external specific surface area according to *Huege et al.*

Applicant is attaching to these Remarks the Declaration of Professor Jean-Paul Pirard, Professor of Chemical Engineering at Liege University, Belgium. Professor Pirard's Resume follows the Declaration. As will be apparent from reading the Declaration of one skilled in the art, and in fact expert in the art of surface area measurements, specific surface area measurements made by the Blaine method referenced in the Huege patent differ greatly from measurements made by the BET method. More particularly, the BET specific surface area is either equal to or higher than the Blaine specific surface area. It will be equal if the particles of the material are strictly non porous, which is not the case with the particles being considered in the present invention. As soon as the particles are slightly porous, the internal surface area of the material becomes important in comparison to the external surface area and consequently, the BET specific surface area is, in general, much larger than the Blaine specific surface area. The accompanying Declaration includes numerical examples of the magnitude of these differences.

Professor Pirard's conclusion is that "the BET specific surface area,  $S_{\text{BET}}$ , and the Blaine specific surface area,  $S_p$ , are two different characteristics of a porous material almost independent of each other. They cannot be taken for one another."

In the last Office Action, the Examiner argues that Applicant's claims "as written do not explicitly recite an internal surface area" and thus the reference still reads on the claims and further that Huege et al. would "broadly encompass the claimed range." Applicant would submit that the claim language

"as measured according to the BET method" should, in and of itself, make a clear distinction in this regard when read in light of the attached Declaration. However, to remove any doubt on this point, Applicant has amended each of independent Claims 1 and 7 to explicitly recite "having particles with a specific surface area, calculated according to the BET method, taking into account internal specific surface area." This language emphasizes the difference between the Blaine method and the BET method, in which the first is related to the external specific surface area, and in which the BET method is related to both the external specific surface area and the internal specific surface area, the internal specific surface area becoming the main specific surface area in the case of porous particles of the type under consideration.

Further, from Table 2 of the Declaration, it can be seen that the BET surface area can be comparable for several different Blaine specific surface areas while, on the contrary that the Blaine specific surface area can be comparable for very different BET specific surface areas. For this reason, it can be seen that the two types of specific surface area are not related one to each other and hence, the BET specific surface area is clearly not linked to particle size as soon as a porous material is involved (as is the case in the present invention).

The attached Declaration also includes comparative data comparing the Huege et al. lime products with products made according to the teachings of the present invention, with a low BET specific surface area. Accordingly, the differences in the teaching of the Huege et al. should be readily apparent.

Based upon the above arguments and amendments, the remaining claims are now thought to be allowable over the art of record, and an early notification of the same would be appreciated.

Respectfully submitted,

Date: September 4, 2009

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## Specific surface area

The specific surface area of a solid material is its surface area by unit of mass. If the mass of a solid is unique, however its surface area is function of the measurement method used. This note aims to compare the signification of the specific surface area determined by the permeability method (Blaine method) and the one determined by the nitrogen adsorption-desorption method (Brunauer-Emmet-Teller method or BET method).

### 1. Blaine method

As far as steady-state permeability in packed powder beds is concerned, any pore space which is located “within” (as distinct from “between”) the particles is of little importance. The external specific surface area, treated here, merely embraces the outer boundary surface area of the solid powder, disregarding its intrinsic porosity. Thus the specific surface area determined with the permeability method represents only the external part of the total specific surface area. The total specific surface area, which takes into account the intrinsic porosity of the solid, is preferably determined by vapour adsorption methods. For the evaluation of catalysts, the entire surface area involved in a reaction is of interest. In mineral processing, the external specific surface area is generally used as a means to characterize the degree of crushing of a material.

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The basic law governing permeability calculations is the Darcy law (1856), which states that the flow rate is proportional to the pressure gradient causing the flow. Kozeny (1927) derived a correlation between permeability, powder bed porosity and particle surface area. This equation was adopted by Carman (1937) for the determination of the specific surface area of powder materials. The specific surface area can be calculated from the permeability, i.e. the time a certain quantity of the permeating fluid needs to flow through a defined powder bed.

Evaluation of the specific surface area can be carried out with the Kozeny-Carman equation for granular beds :

$$S_p = \frac{7d}{\varrho(1-\varepsilon)} \left( \frac{\varepsilon^3 \pi \Delta P}{l \mu Q} \right)^{1/2} \quad [1]$$

where

$S_p$  = specific surface area according to the permeability method ( $\text{m}^2/\text{kg}$ )

$\varrho$  = particle density ( $\text{kg}/\text{m}^3$ )

$\varepsilon$  = porosity of the powder bed (-)

$d$  = bed diameter (m)

$\Delta P$  = pressure drop across the bed (Pa)

$l$  = bed length (m)

$\mu$  = dynamic viscosity of the permeating medium (Pa.s)

$Q$  = flowrate ( $\text{m}^3/\text{s}$ )

The Kozeny-Carman equation [1] which is the basis of the permeability method can be applied to the Blaine technique in the following form :

$$S_p = \frac{C}{\varrho(1-\varepsilon)} \left( \frac{\varepsilon^3 t}{\mu} \right)^{1/2} \quad [2]$$

where

C = apparatus constant

t = running time between two marks on the U-tube manometer

Because in the Blaine technique, the gas volume introduced in the apparatus is constant and the pressure variation is constant, as a consequence the time required to observe the pressure variation is a measure of the permeability and, thus, of the external specific surface area.

The important point is that the porosity  $\varepsilon$  is the powder bed porosity and not the intrinsic porosity of the particles constituting the powder bed. As a consequence, the specific surface area determined by the Blaine method concerns the external surface area of the particles.

## 2. BET method

The BET method, proposed by Brunauer, Emmet and Teller (1938), is based on the following assumptions:

1° the adsorption is localized on well-defined sites, each of them admitting strictly one adsorbed molecule; each site is showing the same energy (homogeneous surface) and the adsorbed molecules do not interact with each other;

2° from the beginning, the adsorption is carried out in several layers, molecules adsorbed in the first layer being adsorption sites for the molecules of the second layer, and so on;

3° there is a permanent equilibrium between the amount of molecules being adsorbed at the surface of the material and the amount of molecules being desorbed from the surface. Desorption is an activated process which activation energy is  $E_1$  for the first adsorbed layer and  $E_L$  for the next layers.  $E_1$  is the heat of adsorption of the molecule at the solid surface and  $E_L$  is the latent heat of liquefaction of the steam at the considered temperature.

The mathematical treatment of those assumptions leads to the equation of the adsorption isotherm, known under the name of BET equation. This equation allows the determination of the vapour volume,  $v_m$ , necessary to cover totally the solid surface area with a monomolecular layer of adsorbate. For nitrogen at a temperature of 77 K,  $v_m$  is directly linked to the BET specific surface area,  $S_{BET}$ , by the relation:

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$$S_{BET} = 4.37 v_m \quad [3]$$

The important point is that nitrogen molecules are adsorbed on the whole accessible surface of the material. As a consequence, the specific surface area determined by the BET method concerns the total surface area of the particles, i.e. the external surface area and the internal surface area, the latter being linked to the intrinsic porosity.

As a conclusion, the BET specific surface area is either equal or higher than the Blaine specific surface area. It will be equal if the particles of the material are strictly non porous. As soon as the particles are slightly porous, the internal surface area of the material becomes important in comparison to the external surface area and, as a consequence, the BET specific surface area is, in general, larger or much larger than the Blaine specific surface area. This conclusion will be highlighted by some examples.

### 3. Examples

Example 1 : Theoretical calculation of the external geometric surface area of a spherical particle of a material.

$$S_{ext} = \frac{6}{d\varrho} \quad [4]$$

where

$d$  = particle diameter (m)

$\varrho$  = particle density ( $\text{kg/m}^3$ ) =  $400 \text{ kg/m}^3$  for standard hydrated lime.

Table 1 : Calculation of the external geometric surface area of a spherical particle of hydrated lime as a function of the particles diameter.

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Particle diameter (m)	$S_{ext}$ ( $m^2/kg$ )	$S_{ext}$ ( $m^2/g$ )
$10^{-3}$ (1 mm)	15	0.015
$10^{-4}$ (100 $\mu m$ )	150	0.15
$5 \times 10^{-5}$ (50 $\mu m$ )	300	0.3
$3 \times 10^{-5}$ (30 $\mu m$ )	500	0.5
$10^{-5}$ (10 $\mu m$ )	1500	1.5
$3 \times 10^{-6}$ (3 $\mu m$ )	5000	5
$1.5 \times 10^{-6}$ (1.5 $\mu m$ )	10000	10
$3.7 \times 10^{-7}$ (0.37 $\mu m$ )	40000	40

Example 2 : Comparison between the Blaine specific surface area and the BET specific surface area measured on 3 samples.

Three samples of hydrated lime were characterised. Sample A is an hydrated lime with a low BET specific surface area,  $S_{BET}$ , sample B is a standard hydrated lime of current industrial production and sample C is an hydrated lime with high BET specific surface area,  $S_{BET}$ . Samples A and B were sieved at 32  $\mu m$  ( $\pm 425$  mesh) in order to highlight the influence of the particle sizes on the specific surface area. The powder retained at 32  $\mu m$  for sample A is noted A>32 $\mu m$  ; the powder passing through is noted A<32 $\mu m$ . Table 2 summarises the values determined at the laboratory for the Blaine specific surface area,  $S_p$ , and for the BET specific surface area,  $S_{BET}$ .

Table 2 : Blaine specific surface area,  $S_p$ , and specific surface area BET,  $S_{BET}$ , of samples A, B and C.

	$S_p$ ( $m^2/g$ )	$S_{BET}$ ( $m^2/g$ )
A	1.1	5.4
A>32 $\mu m$	0.3	5.2
A<32 $\mu m$	1.2	5.6
B	1.3	10.6
B<32 $\mu m$	1.6	9.7
C	1.4	38.8

#### 4. Discussion

At Table 2, it can be seen that the BET specific surface area,  $S_{BET}$ , is systematically at least 5 times larger than the Blaine specific surface area,  $S_p$ . Furthermore, the BET specific surface area,  $S_{BET}$ , for a given hydrated lime is independent of the particles size, while the Blaine specific surface area,  $S_p$ , is dependent of the particle size. In particular, the Blaine specific surface area,  $S_p$ , of the sample A cut with particles size larger than 32  $\mu\text{m}$  ( $0.3 \text{ m}^2/\text{g}$ ) is much lower than the Blaine specific surface area,  $S_p$ , of the not sieved sample A ( $1.1 \text{ m}^2/\text{g}$ ). Conversely, the Blaine specific surface area,  $S_p$ , of the samples cut with particles size lower than 32  $\mu\text{m}$  is always larger than the Blaine specific surface area,  $S_p$ , of the corresponding samples being not sieved. Indeed, at Table 1, it can be observed that the lower the particle size, the larger the geometric external specific surface, corresponding to the Blaine specific surface area.

Table 2 also shows that, for the three samples A, B and C being not sieved, the value of the Blaine specific surface area,  $S_p$ , is very close to one another, while the value of the BET specific surface area,  $S_{BET}$ , may vary with a factor 7, the lowest value being  $5.4 \text{ m}^2/\text{g}$  for sample A and the highest value being  $38.8 \text{ m}^2/\text{g}$  for sample C. This observation means that the BET specific surface area,  $S_{BET}$ , depends in the first degree of factors different than the particle size.

Table 1 and Table 2 also show that the Blaine specific surface area,  $S_p$ , ( $0.3 \text{ m}^2/\text{g}$ ) of the sample A cut with particle size larger than 32  $\mu\text{m}$  ( $A>32\mu\text{m}$ ) is lower than the geometric external specific surface ( $0.5 \text{ m}^2/\text{g}$ ) calculated for a particle diameter of 30  $\mu\text{m}$ ,  $S_{ext}$ . This result is not surprising because the average size of particles of sample cuts with particles size larger than 32  $\mu\text{m}$  is necessarily above 30  $\mu\text{m}$ . The value of Blaine specific surface area of  $0.3 \text{ m}^2/\text{g}$  corresponds to particle diameter of 50  $\mu\text{m}$  in Table 1. On the contrary, the Blaine specific surface area,  $S_p$ , ( $1.2 \text{ m}^2/\text{g}$  et  $1.6 \text{ m}^2/\text{g}$ ), of sample cuts with particle size lower than 32  $\mu\text{m}$  is larger than the geometric external specific surface calculated for a particle diameter equal to 30  $\mu\text{m}$ ,  $S_{ext}$  ( $0.5 \text{ m}^2/\text{g}$ ). The values of the Blaine specific surface area,  $S_p$ , ( $1.3 \text{ m}^2/\text{g}$  et  $1.6 \text{ m}^2/\text{g}$ ) are corresponding to a particle diameter equal to 10  $\mu\text{m}$ ,  $S_{ext}$  ( $1.5 \text{ m}^2/\text{g}$ ).

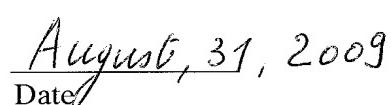
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Finally, at Table 1 and Table 2, it can be observed that if the BET specific surface area,  $S_{BET}$ , would have corresponded to a geometric external specific surface,  $S_{ext}$ , the particle size would vary from 3  $\mu\text{m}$  to 0.37  $\mu\text{m}$  which is quite below the usual particle sizes measured for those materials.

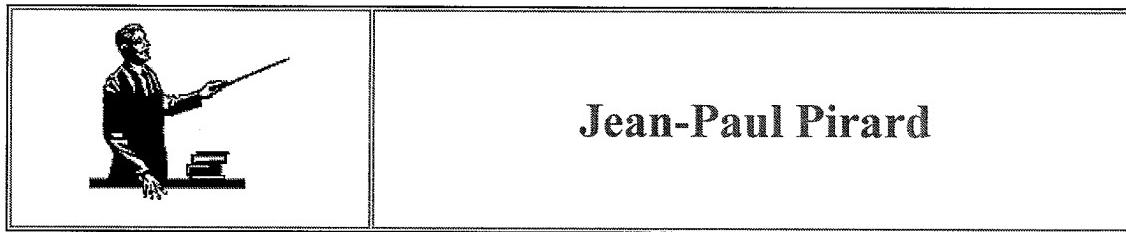
In conclusion, the BET specific surface area,  $S_{BET}$ , and the Blaine specific surface area,  $S_p$ , are two different characteristics of a porous material almost independent of each other. They can not be taken for one another.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

  
Jean-Paul Pirard  
Signature

  
August, 31, 2009  
Date

Jean-Paul PIRARD



**Birth date:** November 16<sup>th</sup> 1947

**Nationality:** Belgian

**QUALIFICATIONS:**

- Chemical Engineer Civil, Liege University, 1970.
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**POSITIONS:**

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**TEACHING DUTIES:**

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- Catalysis (Mass and heat transfers, porous texture ...)

**TECHNICAL EXPERIENCE:**

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1975-1981	Assistant Professor of Chemical Engineering, Liège University
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- Chemical Reactor Engineering.
- Sol-gel process.
- Underground coal gasification
- Porous materials
- Carbon nanotubes

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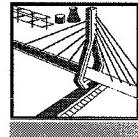


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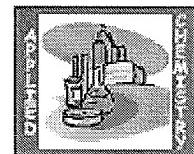


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